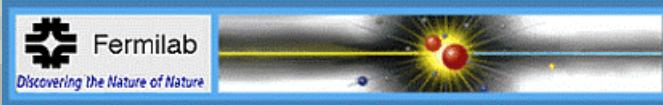




# *Towards higher critical currents in $Nb_3Sn$ superconductors: Recent results from the University of Wisconsin-Madison*

Peter J. Lee (Presenter)

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University of Wisconsin-Madison  
Applied Superconductivity Center

VLHC Magnet Technologies, FNAL, May 24-26, 2000

This Work  
Sponsored by:





# Outline

- How close are we to perfection?
  - A significant increase in the critical current density of Nb<sub>3</sub>Sn is required for both medium and high field VLHC options - are we already close enough to perfection that significant improvements can not be expected?
- Means:
  - Advanced Image Analysis of FESEM images
  - $B^*$  vs  $T$ ,  $J_c$  and  $T_c$  measurements





# **Comparison with a “perfect” superconductor Nb-Ti**

- High energy physics led development of Nb-Ti has resulted in a commodity commercial product available in long lengths at low cost from a number of highly competitive companies.
  - With the exception of a small region at the edge of the filaments the microstructure is homogeneous across each filament.
  - The micro-structure/chemistry is uniform from filament to filament.
  - The filaments are uniform in shape and size and are uncoupled.
  - The role of chemistry, heat treatment and processing on the  $J_c$  and *why* is well understood and well controlled.





## ***How does Nb<sub>3</sub>Sn compare?***

- We are in the process of characterizing a variety of Nb<sub>3</sub>Sn strands available in commercial quantities - they are all excellent strands
  - but none are perfect . . .
- Two examples of high performance Nb<sub>3</sub>Sn
  - MJR style internal Sn (high Sn)
  - SMI-PIT
- A low-Sn ITER strand.



Critical Current  
Density, A/mm<sup>2</sup>

100,000

10,000

1,000

100

At 4.2 K Unless  
Otherwise Stated

**YBCO**

**YBCO**  
**75 K H||a-b**

**2212**

0

5

10

15

20

25

Applied Field, T

2223

75 K H||c

**VLHC Magnet Technologies, FNAL, May 24-26, 2000**

University of Wisconsin-Madison  
Applied Superconductivity Center

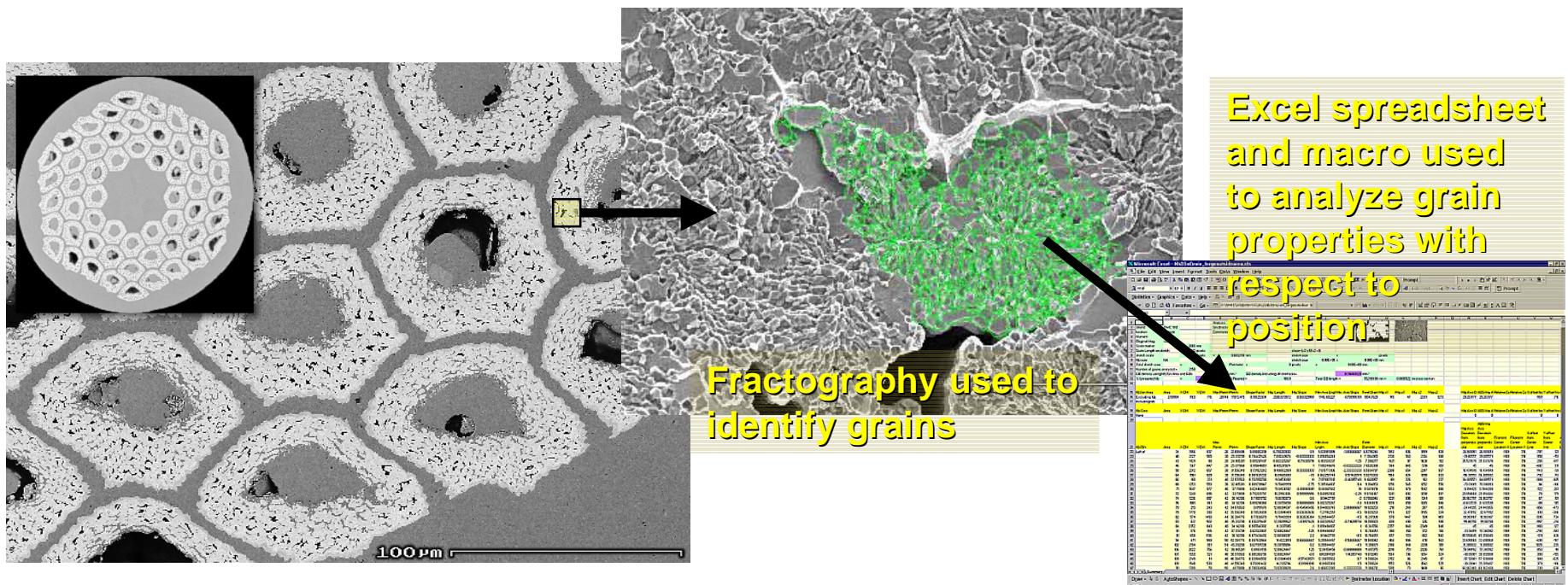


- Nb-Ti: Nb-Ti/Nb (21/6) 390 nm multilayer '95 (5°), 50 μV/cm - McCambridge et al. (Yale)
- Nb-Ti: Nb-Ti/Ti (19/5) 370 nm multilayer '95 (0°), 50 μV/cm - N. Rizzo et al. LTSC'96 (Yale)
- Nb-Ti: APC strand Nb-47wt.%Ti with 24 vol.%Nb pins (24 nm nominal diam.) - Heussner et al. (UW-ASC)
- Nb-Ti: Aligned ribbons, B|| ribbons, Cooley et al. (UW-ASC)
- Nb-Ti: Best Heat Treated UW Mono-Filament. (Li and Larbalestier, '87)
- Nb-Ti: Example of Best Industrial Scale Heat Treated Composites -1990 (compilation)
- Nb-Ti(Fe): 1.9 K, Full-scale multifilamentary billet for FNAL/LHC (OS-STG) ASC'98
- Nb-Ti: Nb-47wt%Ti, 1.8K, Lee, Naus and Larbalestier (UW-ASC'96) ICMC-CEC1997.
- Nb-44wt%Ti-15wt%Ta: at 1.8K, monofil. optimized for high field, unpub. Lee, Naus and Larbalestier (UW-ASC'96)
- Nb<sub>3</sub>Sn: Internal Sn High J<sub>c</sub> design CRE1912, OI-STG, - Zhang et al. ASC'98 Paper MAA-06
- Nb<sub>3</sub>Sn: Internal Sn High J<sub>c</sub> design ORe0038, OI-STG, - Zhang et al. ASC'98 Paper MAA-06
- Nb<sub>3</sub>Sn: Internal Sn, ITER type low hysteresis loss design - (IGC - Gregory et al.) [Non-Cu J<sub>c</sub>] ,
- Nb<sub>3</sub>Sn: Bronze route int. stab. -VAC-HP, non-(Cu+Ta) J<sub>c</sub>, - Thoner et al., Erice '96.
- Nb<sub>3</sub>Sn: SMI-PIT, non-Cu J<sub>c</sub> 10 μV/m, 192 fil., 1 mm dia. (45.3 % Cu), - U-Twente data provided March 2000 by SMI
- Nb<sub>3</sub>Sn: Tape from (Nb,Ta) <sub>1-x</sub>Nb<sub>x</sub>+Nb-4at.%Ta powder, [Core J<sub>c</sub>, core -25 % of non-Cu area] Tachikawa et al. (Tokai U.), ICMC-CEC '99
- Nb<sub>3</sub>Al: 84 Fil. RHQT Nb/Al-Mg(0.6 μm), - Iijima et al. NRIM ASC'98 Paper MVC-04
- Nb<sub>3</sub>Al: 84 Fil. RHQT Nb/Al-Ge(1.5 μm), - Iijima et al. NRIM ASC'98 Paper MVC-04
- Nb<sub>3</sub>Al: Nb stabilized 2-stage JR process (Hitachi,TML-NRIM, IMR-TU), Fukuda et al. ICMC/ICEC '96
- Nb<sub>3</sub>Al: Transformed rod-in-tube Nb<sub>3</sub>Al (Hitachi,TML-NRIM), Nb Stabilized - non-Nb J<sub>c</sub>, APL, vol. 71(1), p.122, 1997
- YBCO: /Ni/YSZ ~1 μm thick microbridge, H||c 4 K, - Folty et al. (LANL) '96
- YBCO: /Ni/YSZ ~1 μm thick microbridge, H||ab 75 K, - Folty et al. (LANL) '96
- YBCO: /Ni/YSZ ~1 μm thick microbridge, H||c 75 K, - Folty et al. (LANL) '96
- Bi-2212: 3-layer tape (0.15-0.2 mm 4.0-4.8 mm) B||tape at 4.2 K face - Kitaguchi et al. ISS'98, 1 μV/cm
- Bi-2212: paste, B||tape, 4.2 K - Hasegawa et al. (Showa) IWS'95
- Bi-2212: stack, B||tape, 4.2 K - Hasegawa et al. (Showa) IWS'95
- Bi-2212: 19 filament tape B||tape face - Okada et al (Hitachi) '95
- Bi-2212: Round multifilament strand - 4.2 K - (IGC) Motowidlo et al. ISTE/MRS '95
- Bi-2223: multi, B||tape, 4.2 K - Hasegawa et al. (Showa) IWS'95
- Bi-2223: Rolled 85 Fil. Tape, B||, - (AmSC) UW'6/96
- Bi-2223: Rolled 85 Fil. Tape, B ⊥, - (AmSC) UW'6/96
- PbSnMo<sub>6</sub>S<sub>8</sub> (Chevrel Phase): Wire with 20%SC in 14 turn coil, - (Univ. Geneva/HFML/RIM - NL/U-Rennes), 97



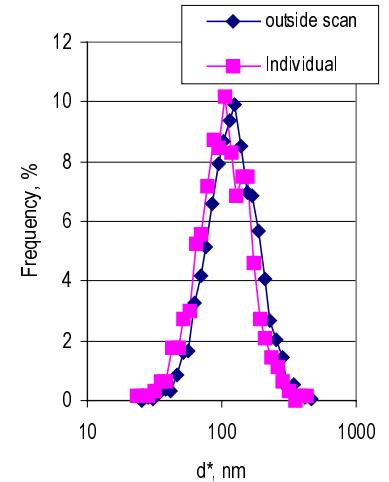
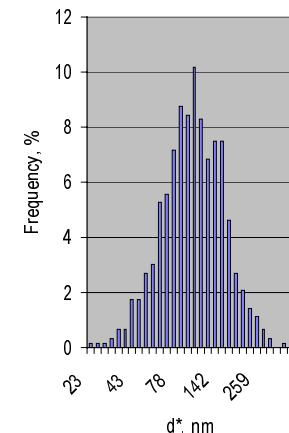
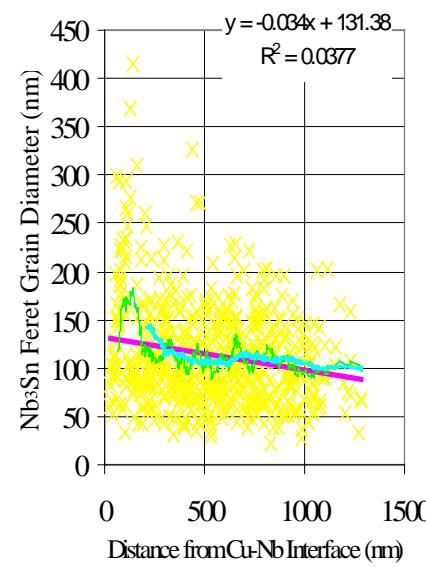
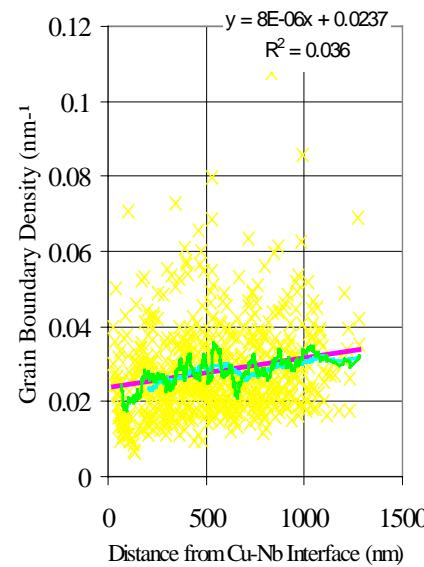
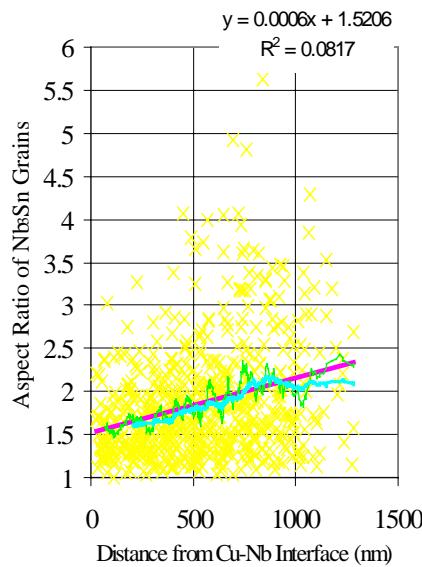
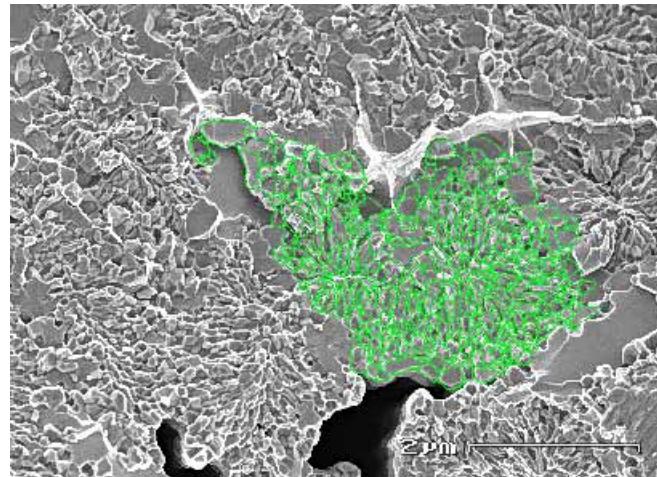
## Example I: High Sn MJR

- Relatively good fabricability (strand and cable) and cost.
- Limit to Sn content available in package.





# TWC 1912 Single filament

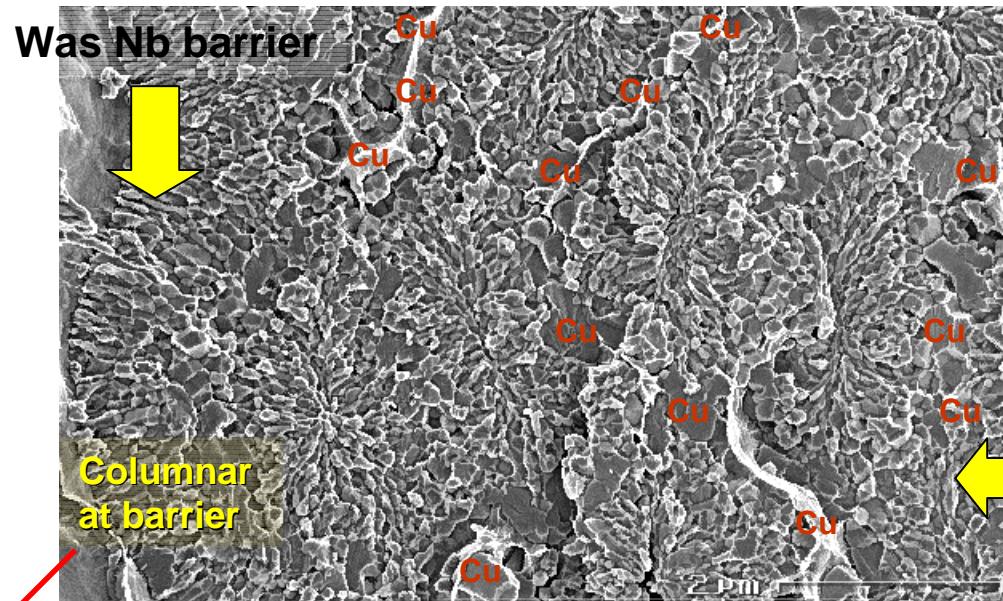
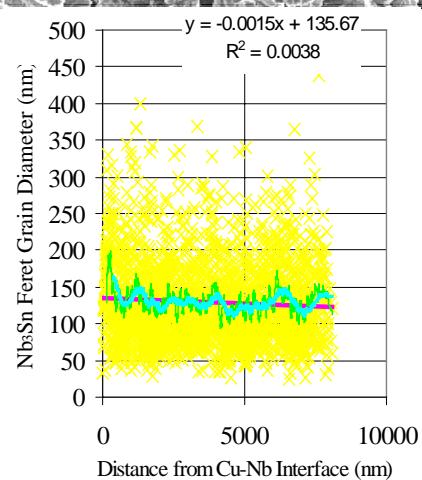
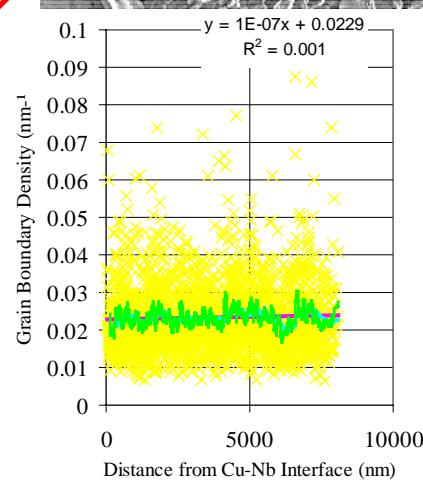
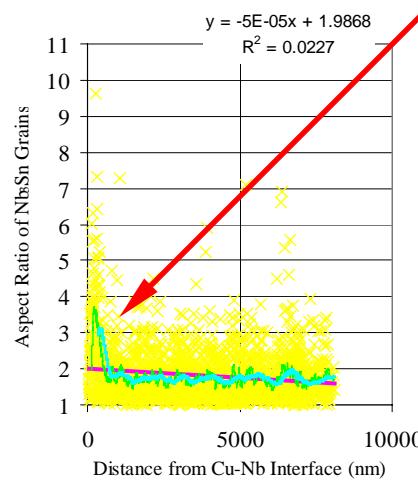
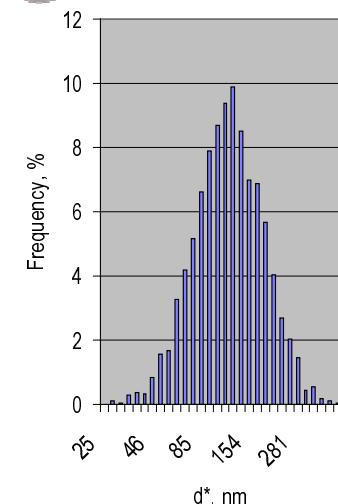


Even with  
densely packed  
filaments  
morphology  
variations occur  
across individual  
filaments.





# CRe1912 overall $\mu$ -structure



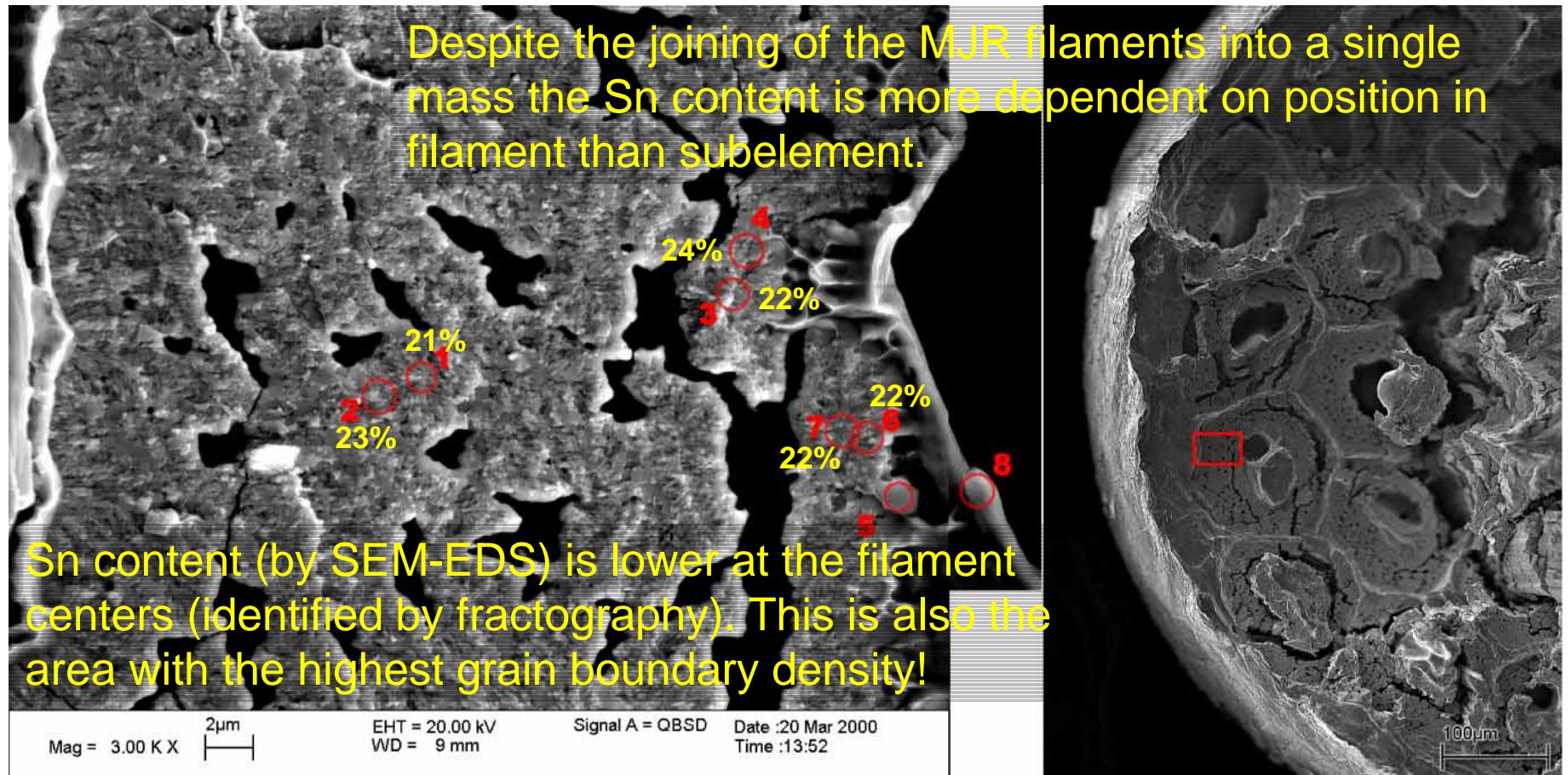
Was Sn core  
Was Nb-1%Ti

Apart from the columnar grains at the barrier there is a continuous but small morphological variation across the layer in grain boundary density.





# Variation in Sn content of A15 OI-ST High $J_c$ MJR Strand



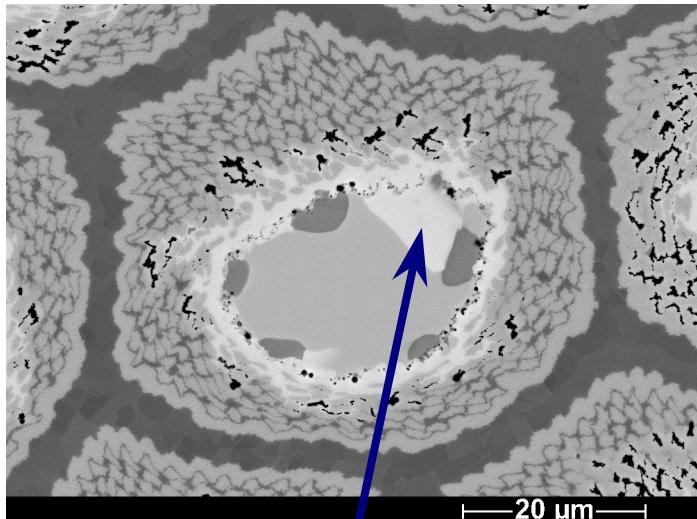
Note: The % numbers are an average over the region represented by the red circle. The true range can be expected to be higher



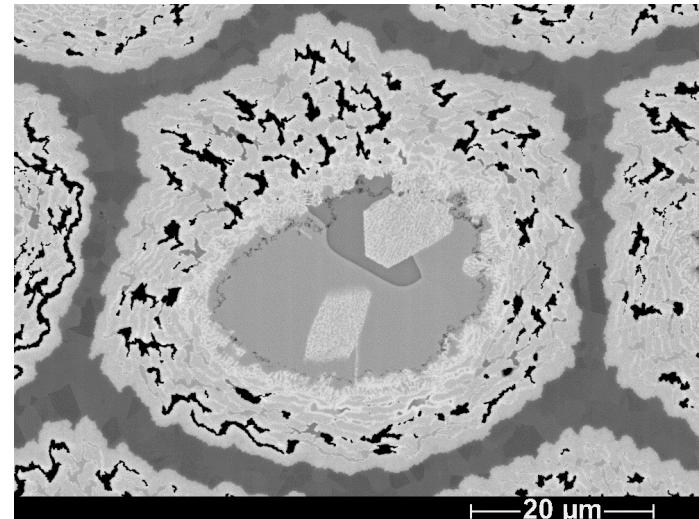


# Cu-Sn-Nb Phases - High Sn MJR

150 hours / 362° C



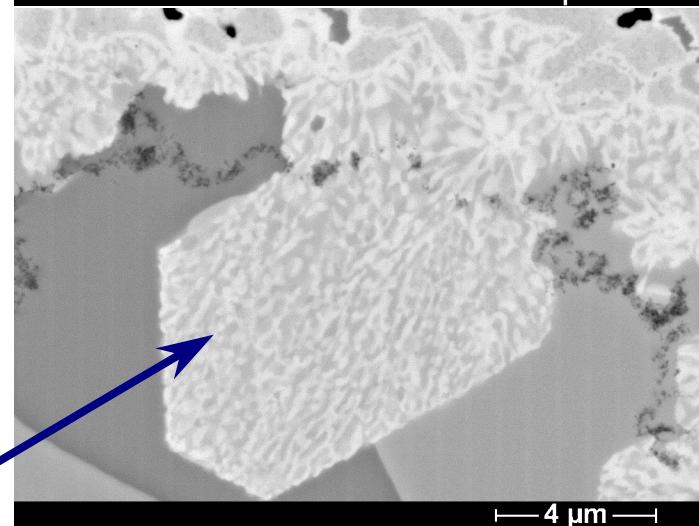
150 hours / 510° C



Cu-23at.%Nb-62at.%Sn

We have an unexpected movement of Nb to the center.

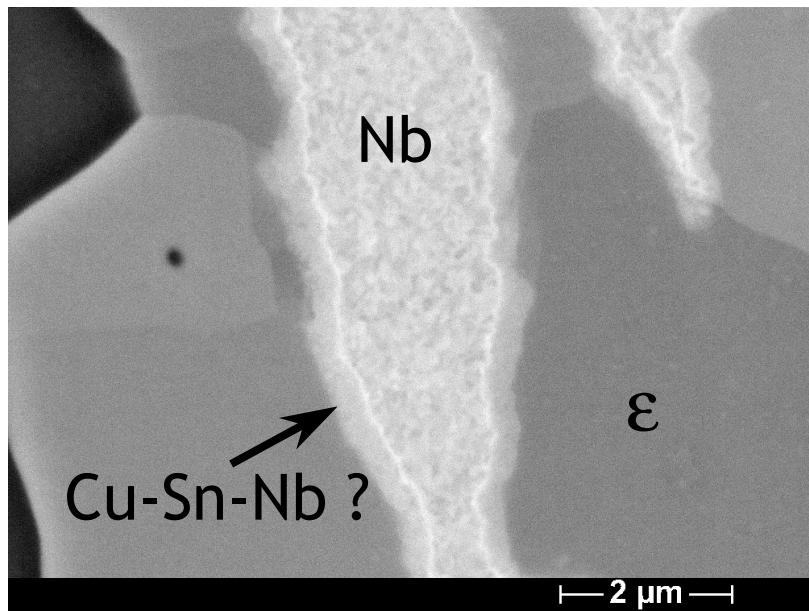
Cu-20at.%Nb-30at.%Sn



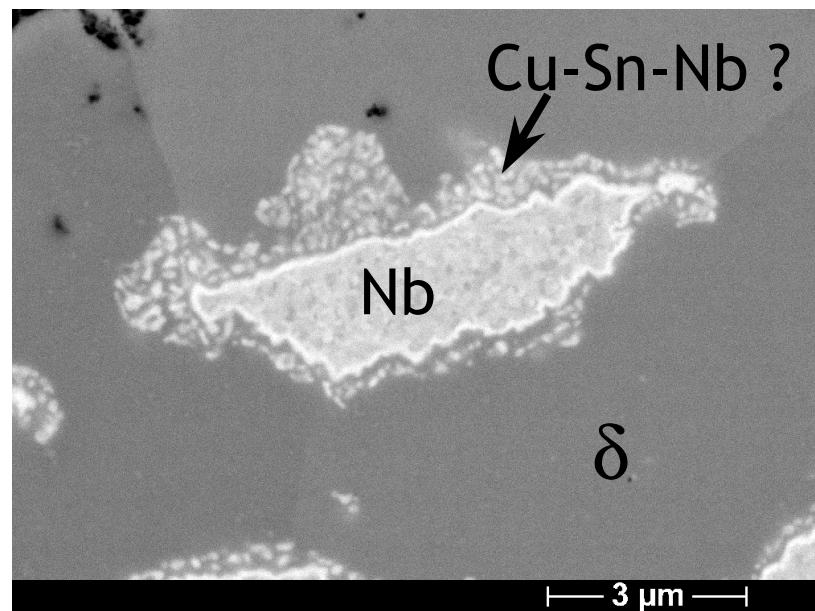


# Cu-Sn-Nb Phases - Low Sn Strand

24 hours / 401° C



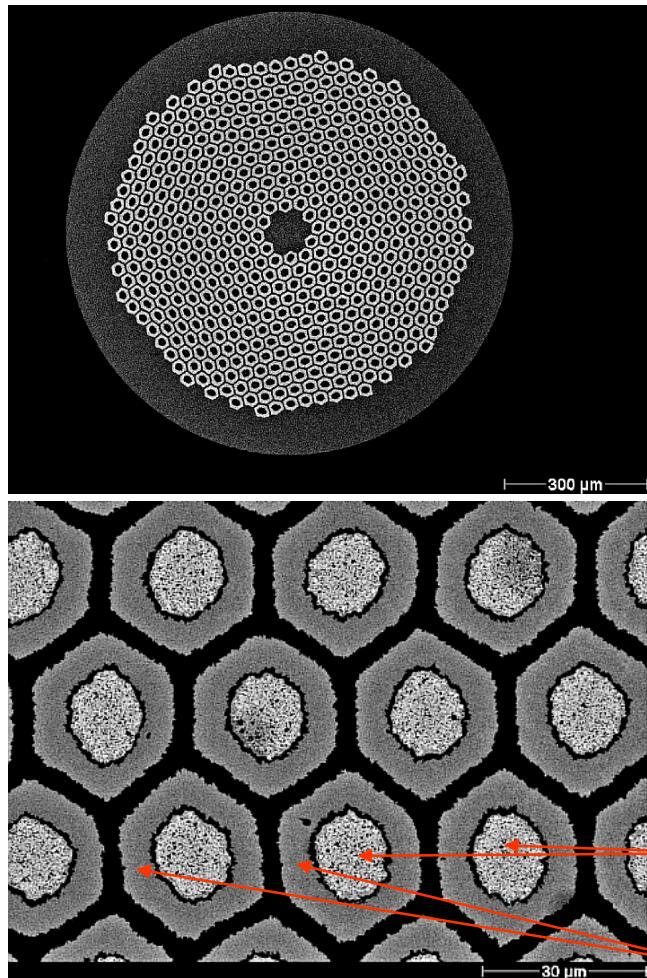
150 hours / 510° C





## Example II:

### Non-Alloyed SMI-PIT Strand



- High Sn concentration available from  $\text{NbSn}_2$
- Efficient Non-Cu package with no “wasted” Cu.
- High Field Nb(Ta) based strand now available.
  - Nb $\text{Sn}_2$  powder + Cu powder core
  - Nb tubes

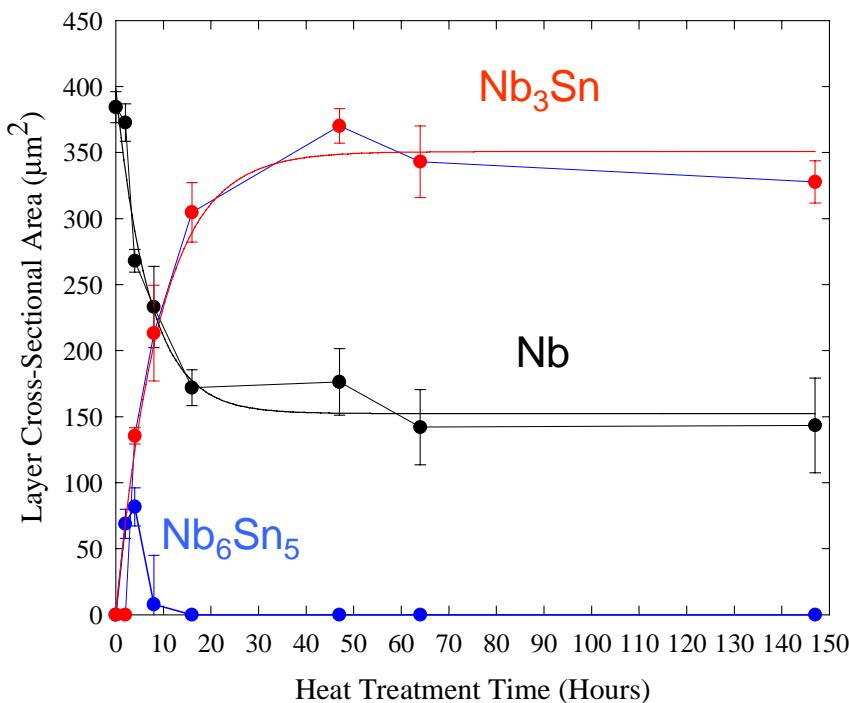




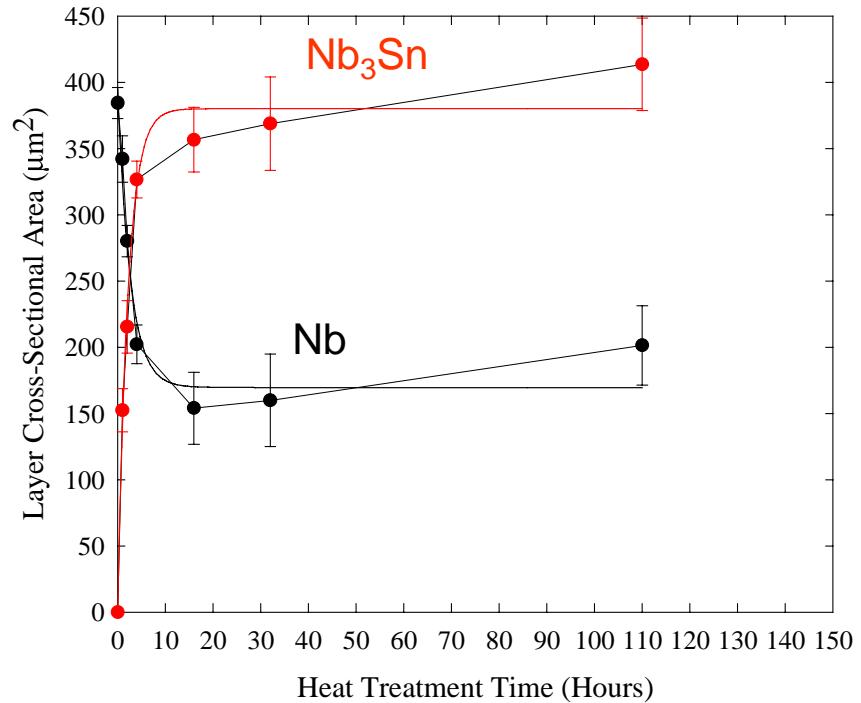
# ***Nb<sub>3</sub>Sn Layer Growth is Fast***

After 47 hour HT S/C x-section: 0.19 mm<sup>2</sup> Compared to 0.13 mm<sup>2</sup> for internal Sn and 0.05 mm<sup>2</sup> for bronze x-sections

675 °C HT



750 °C HT



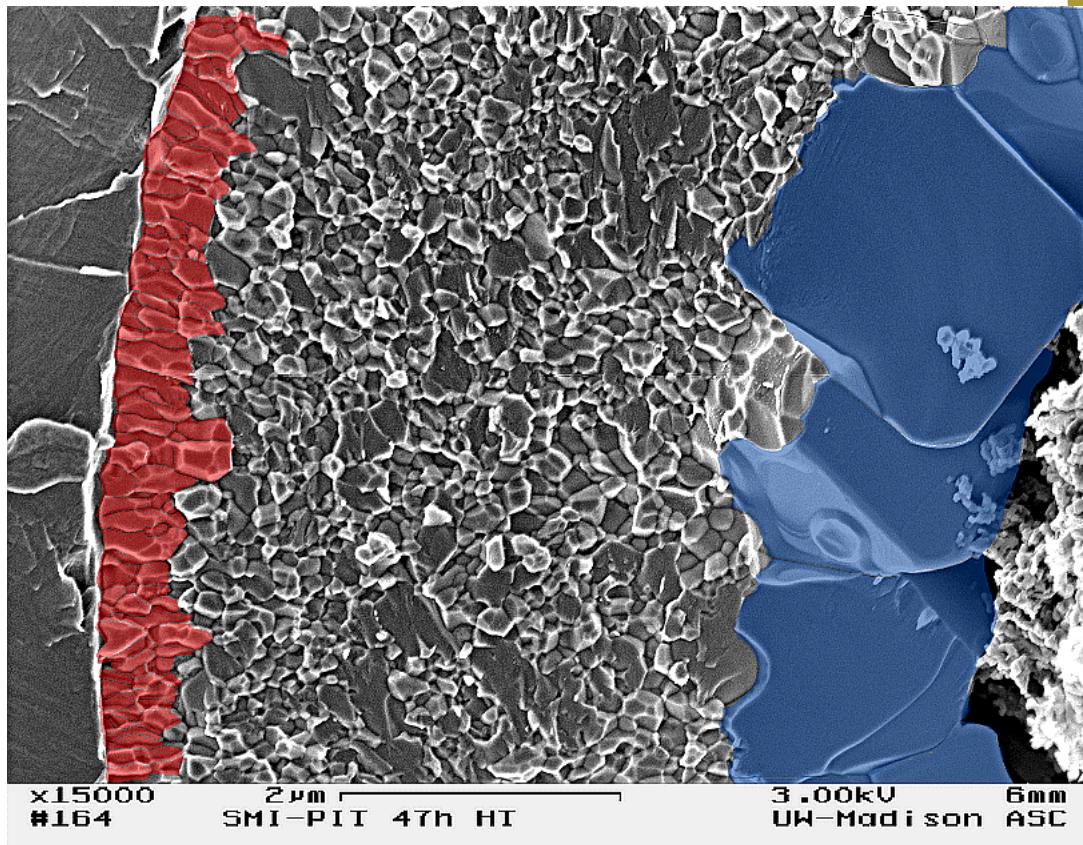
Growth rate flattens between 32 and 47 hrs.

Growth rate flattens between 8 and 10 hrs.





# Analysis of Layer $J_c$ and $Q_{gb}$ in non-alloyed SMI-PIT strand



In this partial fracture cross-section of a SMI-PIT strand, three distinct grain size/morphology regions are observed.

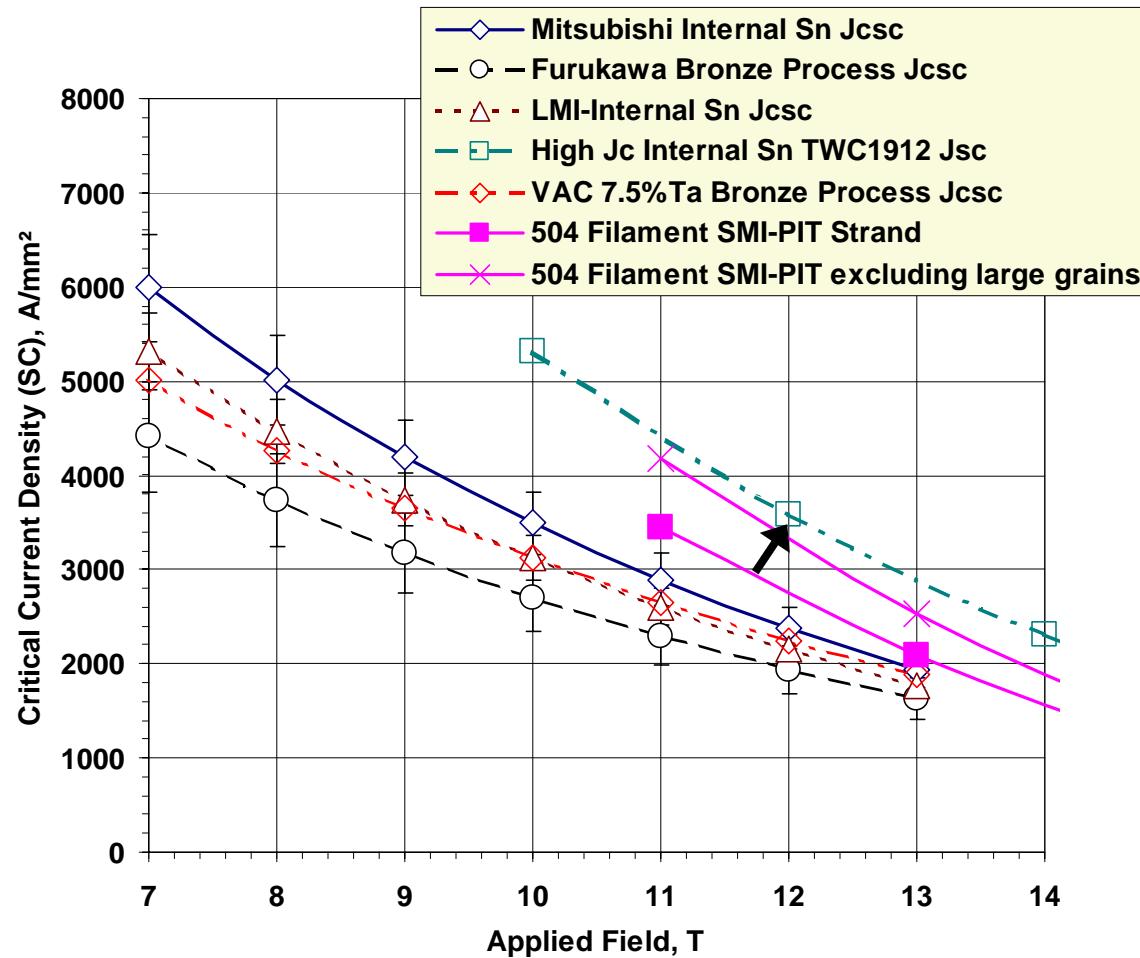
- Columnar adjacent to the Nb
- Fine equiaxed center
- Very large grains adjacent to core.

If the very low grain boundary density in the large-grain area means that this area does not contribute significantly to  $I_c$  then we can adjust the  $J_{csc}$  data to reflect the “good” area.





# Non-Alloyed SMI PIT $J_{csc}$



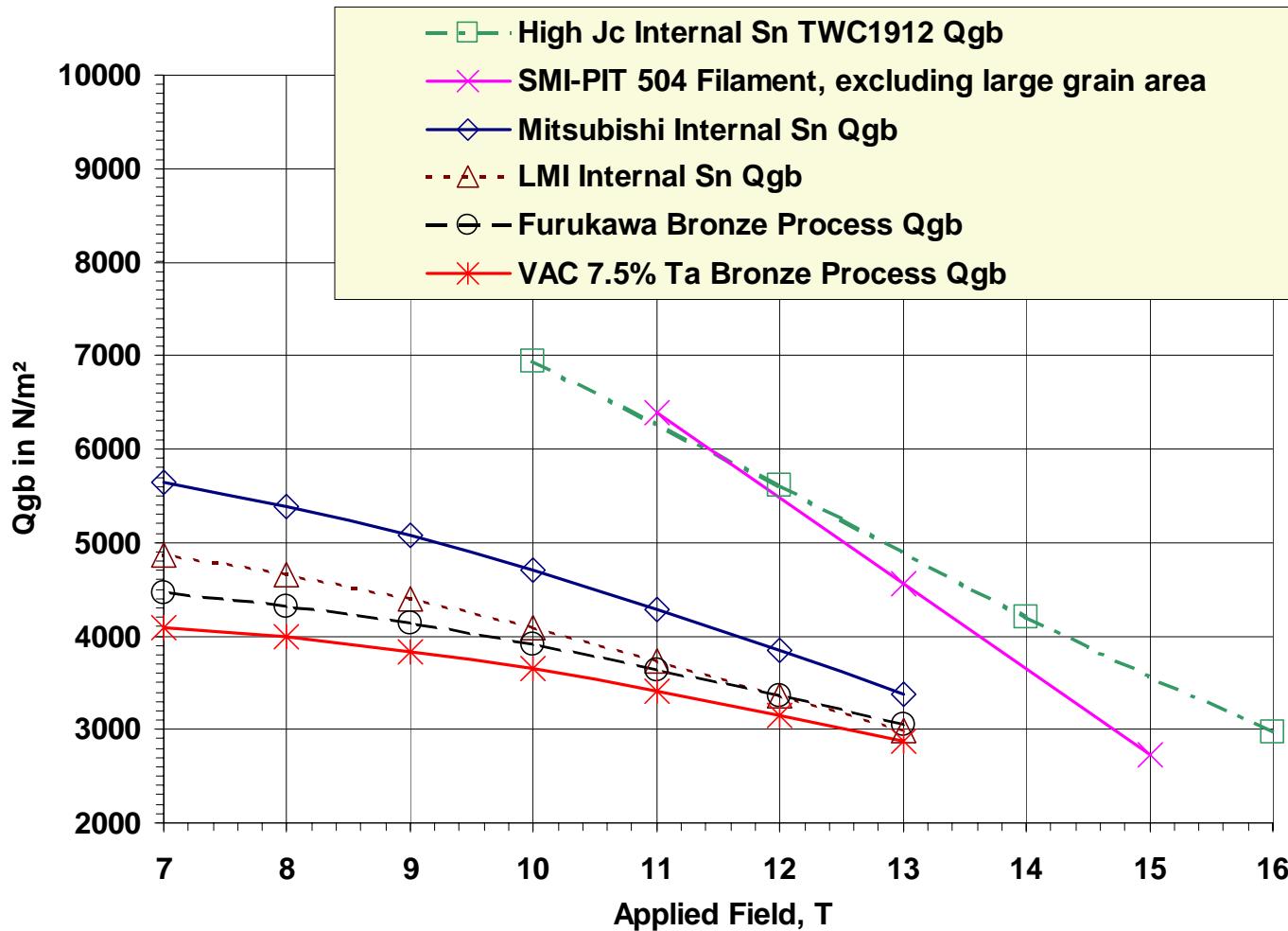
When the  $J_{csc}$  data is calculated excluding the non-contributing large grain area the performance of the PIT A15 more closely matches the high  $J_{csc}$  internal Sn strand.

The higher  $J_c$  of the new Nb(Ta)<sub>3</sub>Sn can be expected to increase the  $J_{csc}$  further





# Specific Grain Boundary Pinning Comparison



High Sn and PIT strands have 50% higher  $Q_{gb}$  (and  $J_{csc}$ ) than ITER style strands

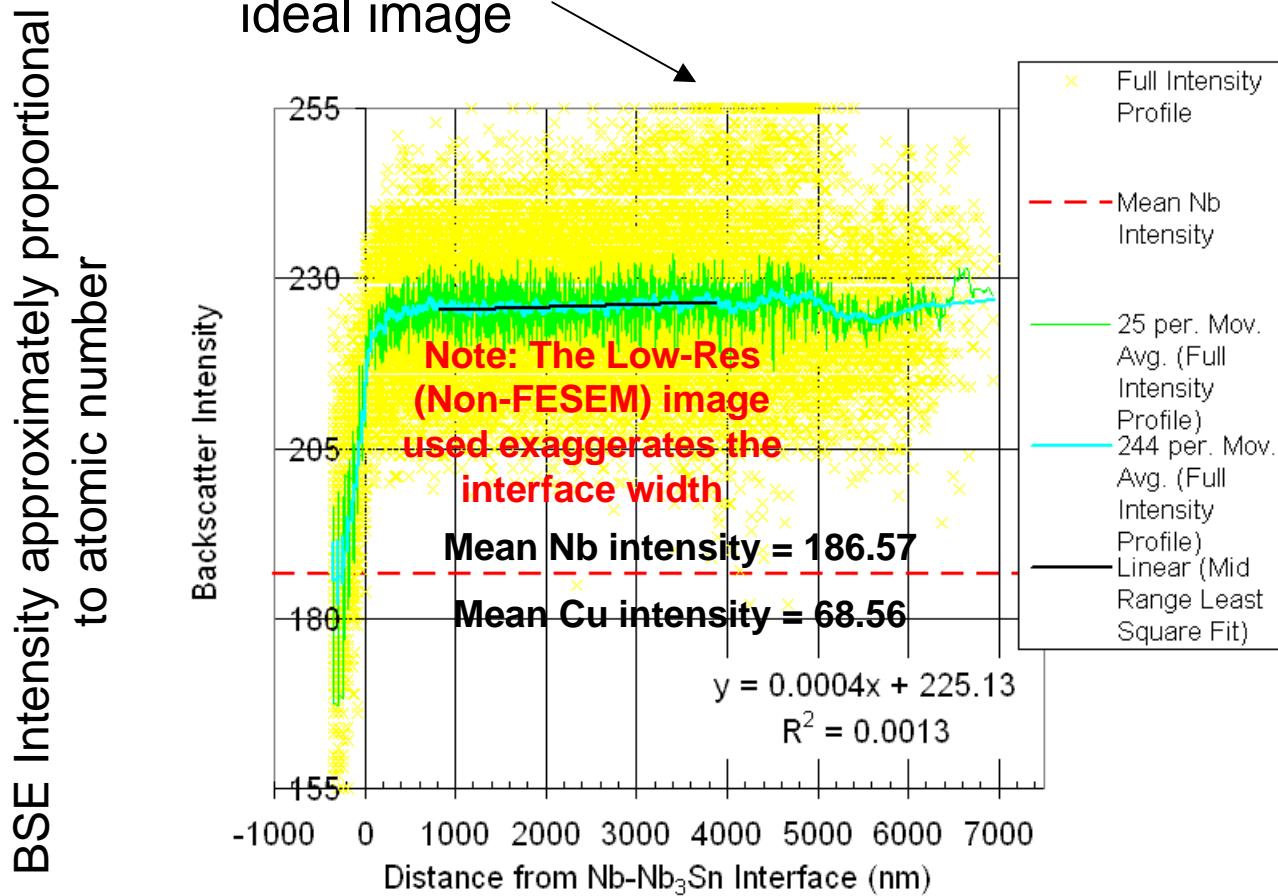
The new  $Nb(Ta)_3Sn$  PIT should not have the high field tail off observed here.



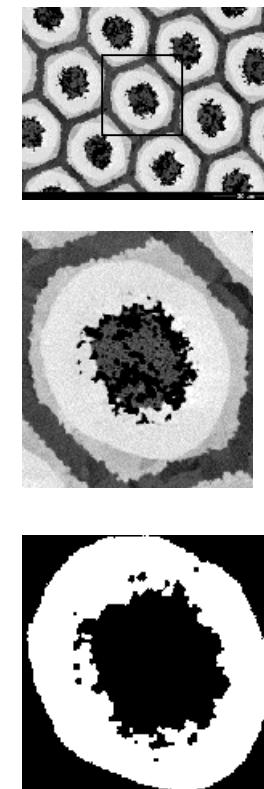


# Composition analysis of SMI PIT

Saturation . . Not an ideal image

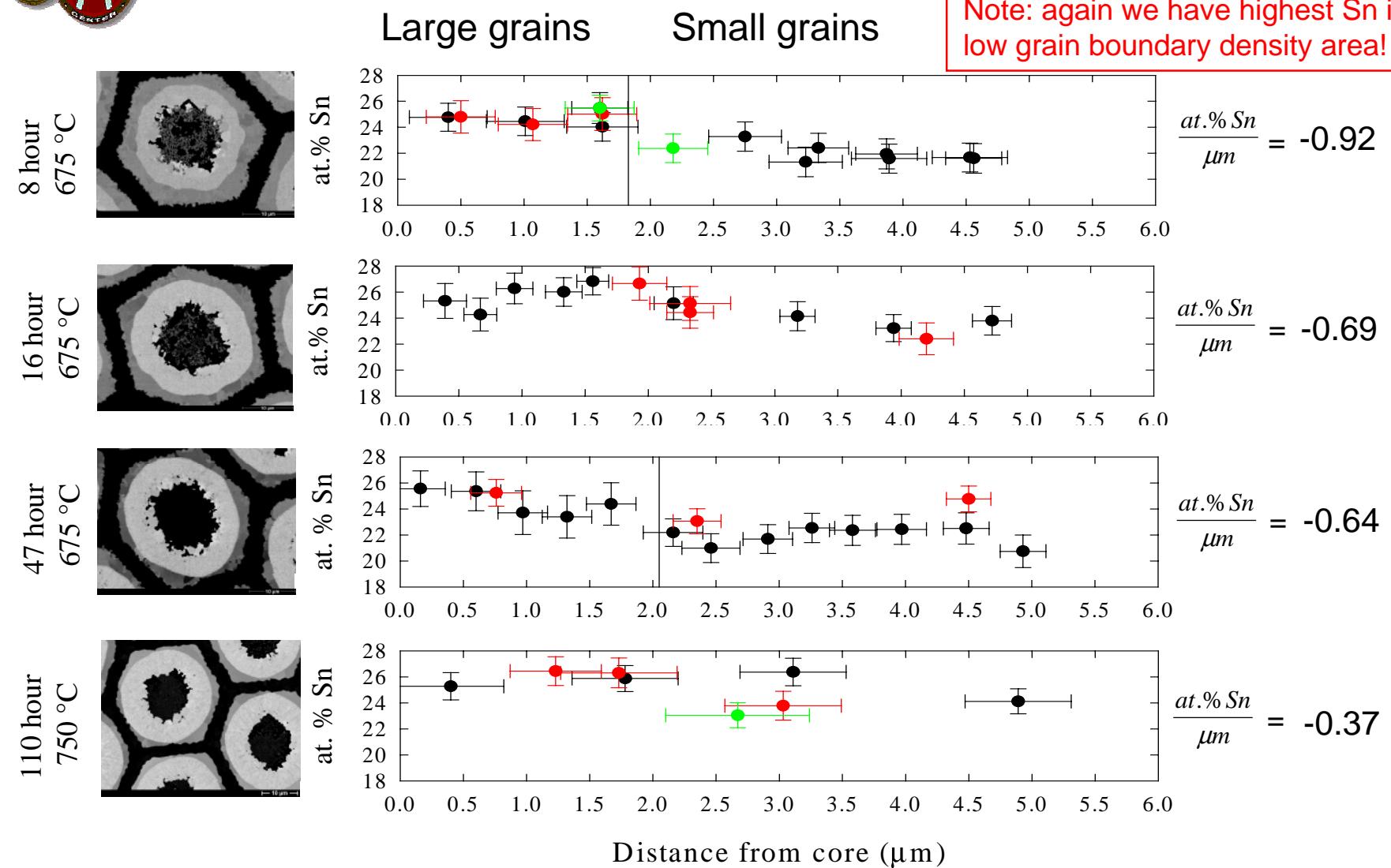


BSE technique: P. J. Lee and D. C. Larbalestier, Microscopy & Microanalysis 2000.





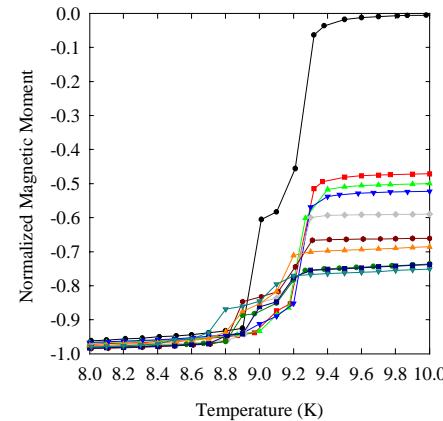
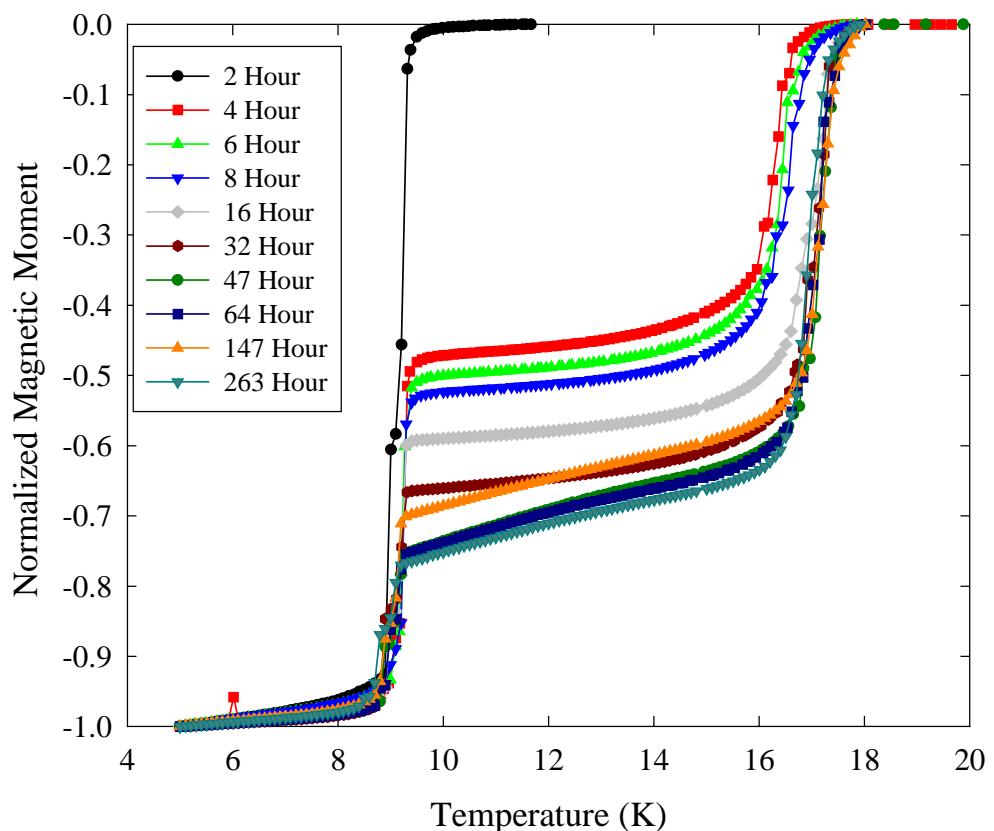
# EDX Measurements





# Inductive $T_c$ Measurements

## 675 °C HT $T_c$ Results

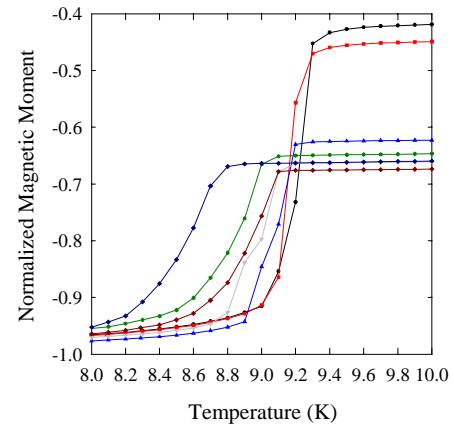
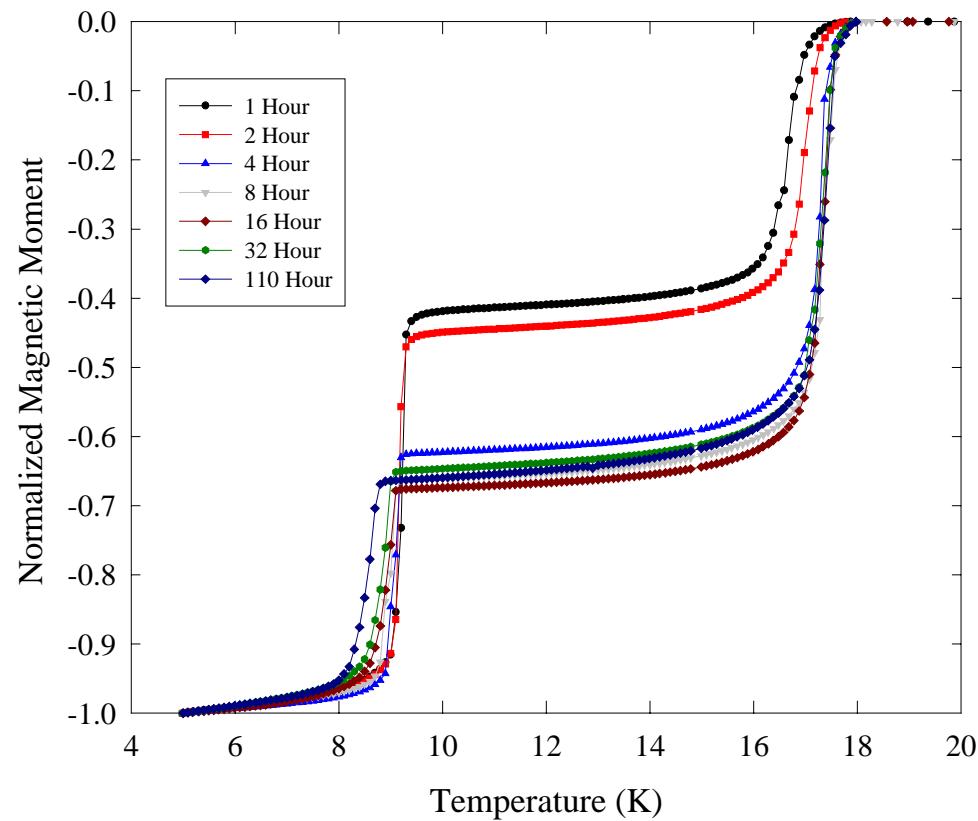


- Steady rise in  $\text{Nb}_3\text{Sn}$   $T_c$  with HT time.
- “Nb transition” decreases and broadens with HT time.
- $\text{Nb}_3\text{Sn}$  layer thickening.





# 750 °C HT $T_c$ Results

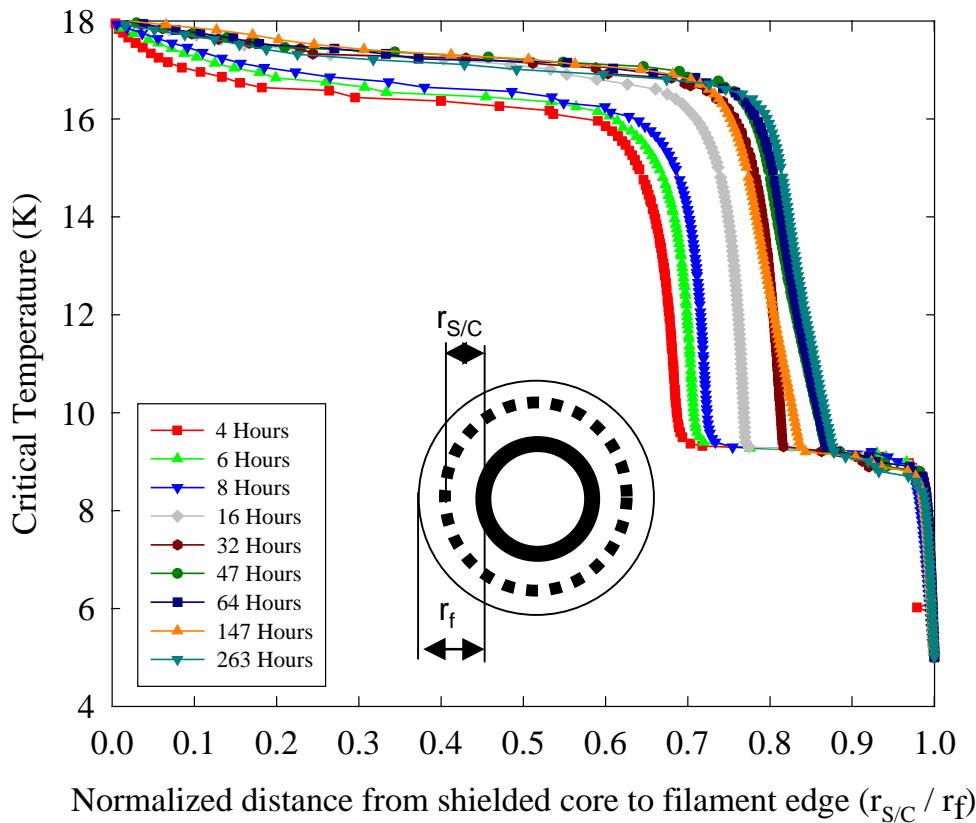


● Similar to 675 °C results except faster.





# 675 °C $T_c$ Profiles

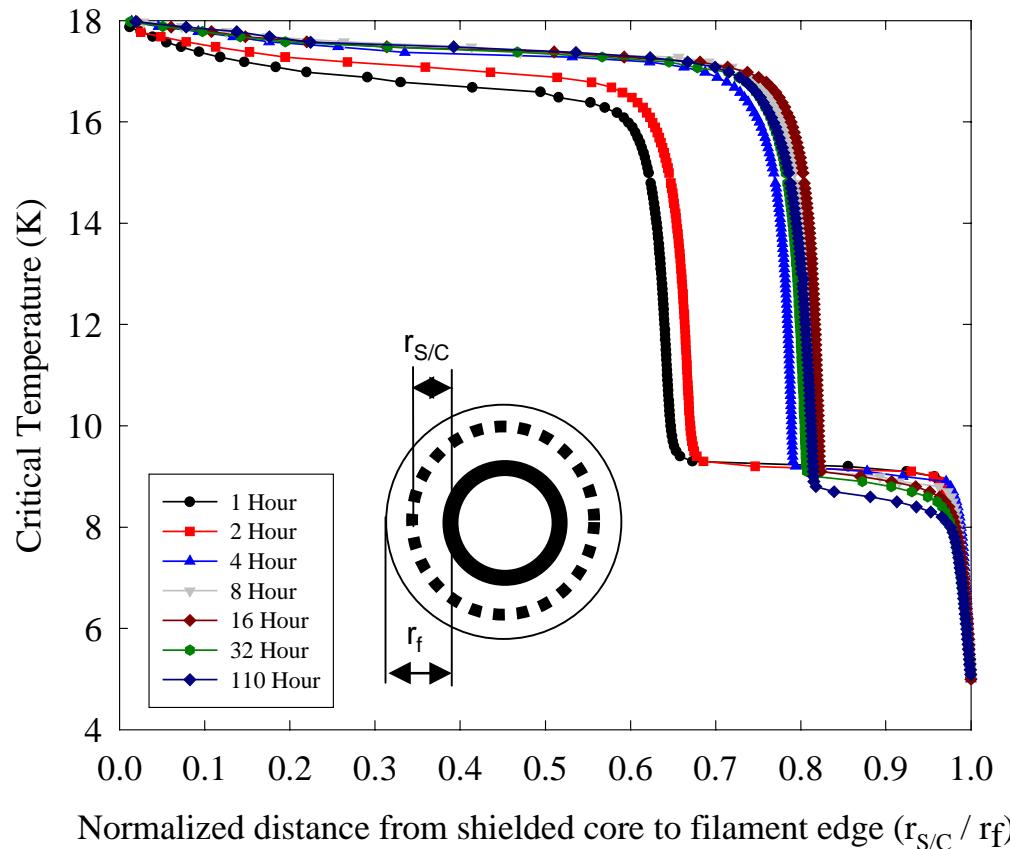


- 4-8 hour plots have a steeper slope.
- Layer growth visible.
- After 32 hours growth rate slows.
- Tail appears at ~ 14 K after 47 hour HT.





# 750 °C $T_c$ Profiles



- 1 and 2 hour plots have a steeper slope.
- After 4 hours growth rate slows.
- No tail appears at  $\sim 14$  K.





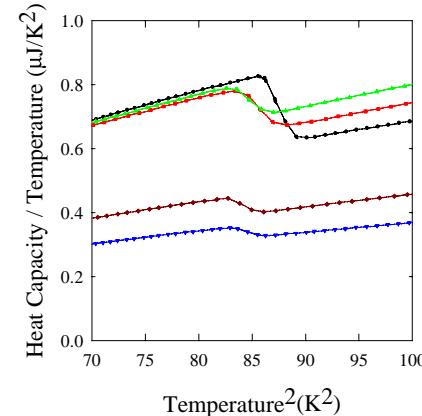
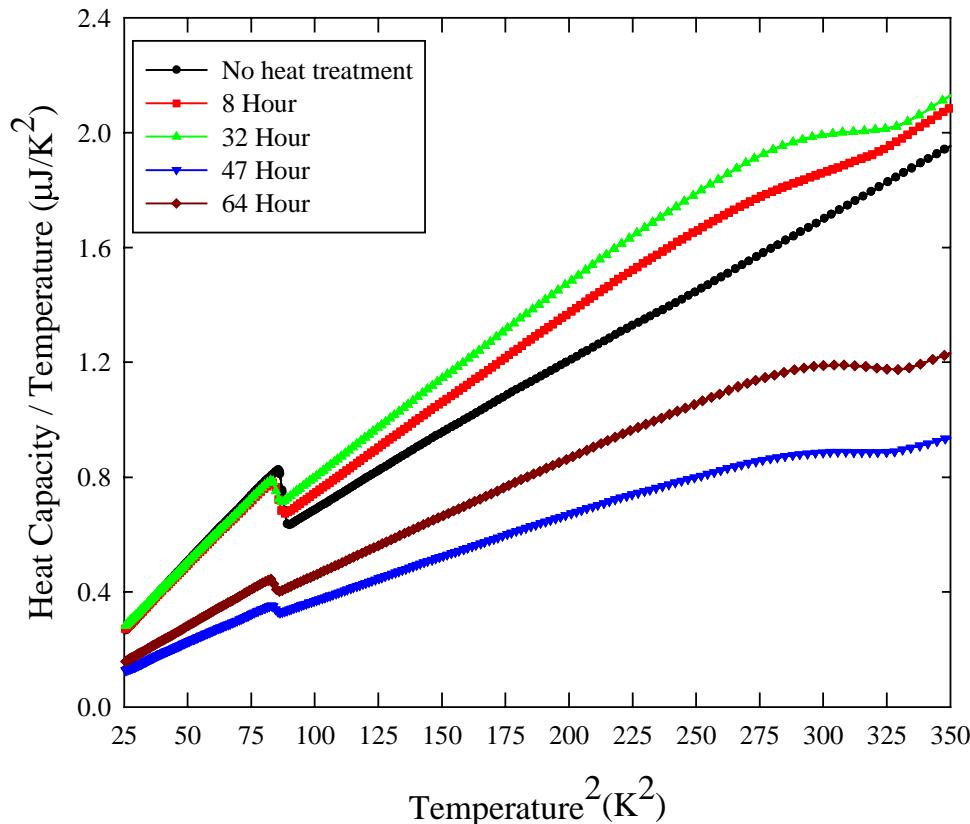
# *Inductive Critical Temperature and EDX Analysis Summary*

- EDX reveals:
  - Majority of layer ~ 20 - 25 at.%Sn
  - Heat treatment improves overall layer composition.
  - But highest Sn concentration in large grain region
- Inductive  $T_c$  measurements reveal:
  - $T_c$  range across majority of the layer: ~ 15.5 -18.3 K.
  - $T_c$  gradient flattens and increases with HT time.
  - $T_c$  drops rapidly at the interface of Nb<sub>3</sub>Sn-Nb and Nb<sub>3</sub>Sn-Cu.
- Gradient from EDX and inductive measurements: 0.99 - 1.67 K/at.%Sn





# 675 °C Heat Capacity Results

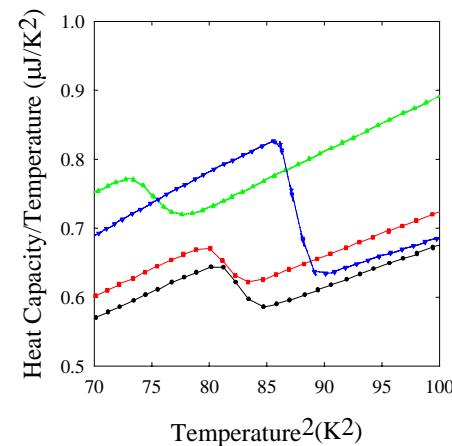
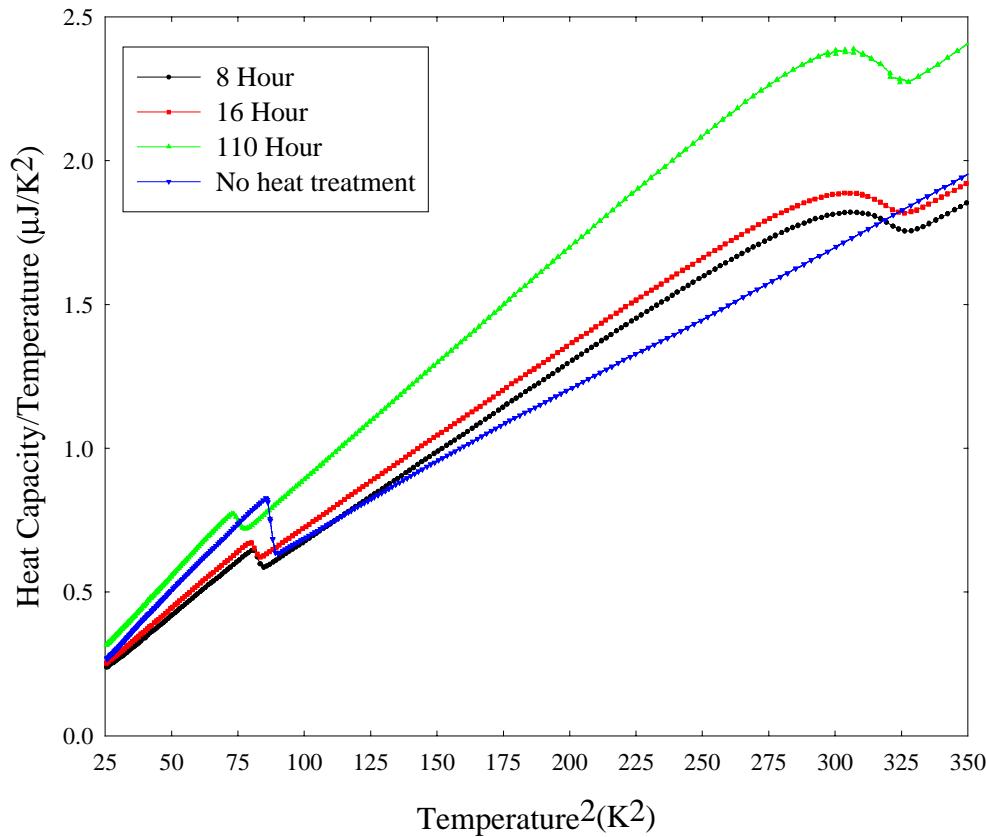


- Differences in magnitudes due to differences in sample's masses.
- $\text{Nb}_3\text{Sn}$  transition becomes more pronounced with HT time.
- “Nb transition” reaffirms inductive  $T_c$  measurement.





# 750 °C Heat Capacity Results

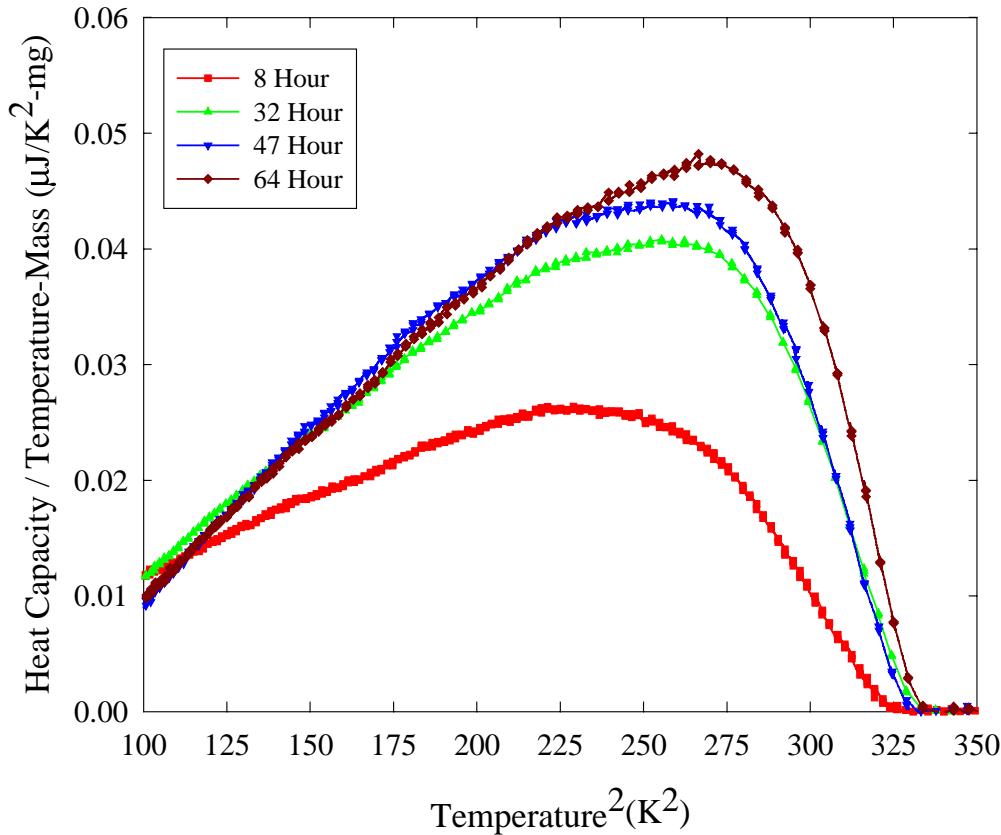


- Similar to 675 °C results except faster.





# 675 °C Heat Capacity Results

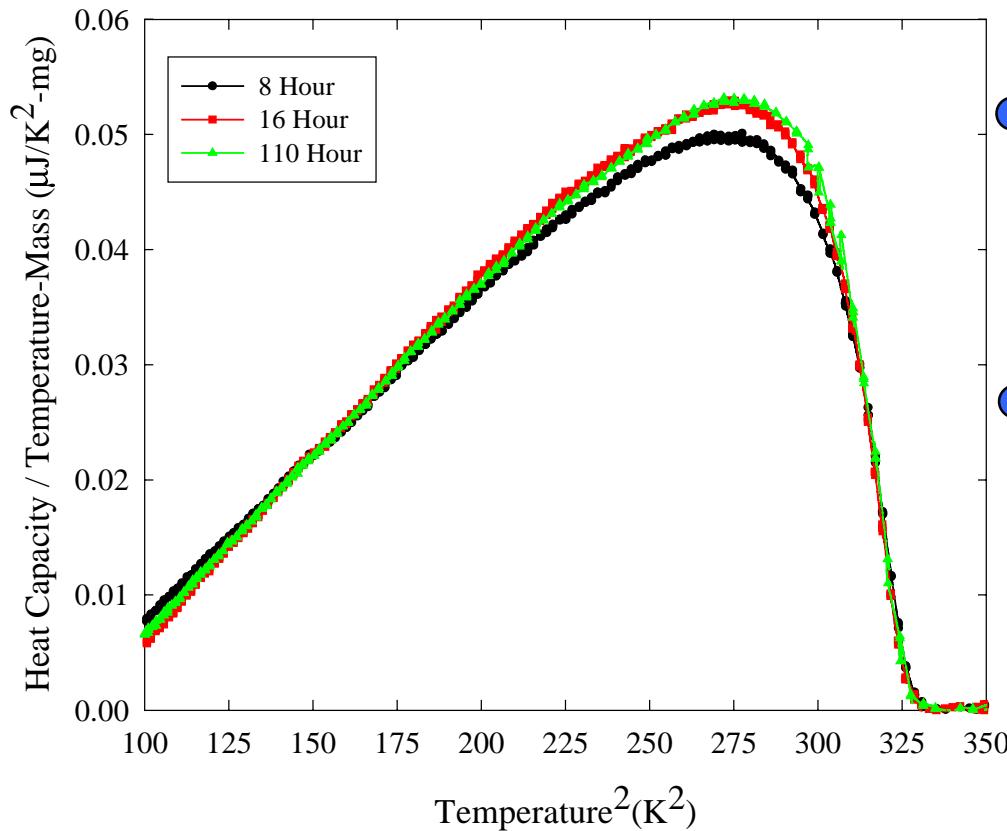


- Increasing HT time results in:
  - Sharper transition.
  - Increase in max  $T_c$ .
  - Increase in the amount of  $\text{Nb}_3\text{Sn}$ .





# 750 °C Heat Capacity Results



- After 8 hour HT:  
 $\text{Nb}_3\text{Sn}$  layer as thick as 64 hour.  
Transitions are sharp.
- HC measurements coincide  
well with SEM-EDX/BSE  
measurements





# ***Non-Alloyed SMI-PIT and “Perfection”***

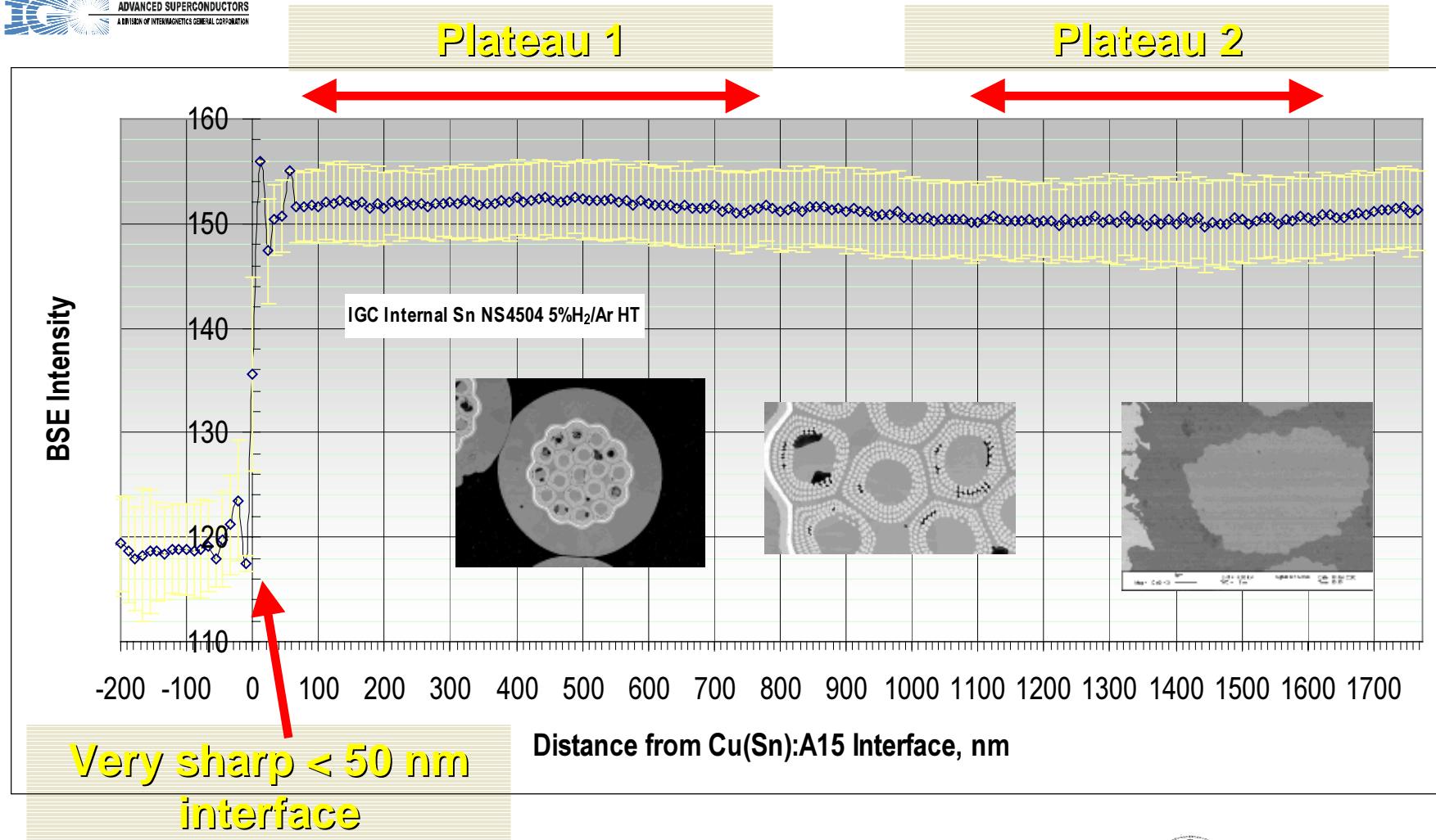
- Current heat treatments limited by potential Sn diffusion into Cu.
- The gradient in most of the layer is flat and contains high quality, high  $T_c$  material ( $J_c$  (non-Cu @ 12T) = 1690 A/mm<sup>2</sup>)  
*but the highest Sn is in the large grain region.*
- Inductive  $T_c$  measurements:  
Semi-quantitative/qualitative information about the layer growth and the  $T_c$  gradient.  
Changes in composition gradient and  $T_c$  gradient in agreement.
- Heat Capacity Measurements:  
Information about the quality of the superconducting material.  
Relative amounts of material.  
Results agree with inductive  $T_c$  and SEM/EDX measurements.



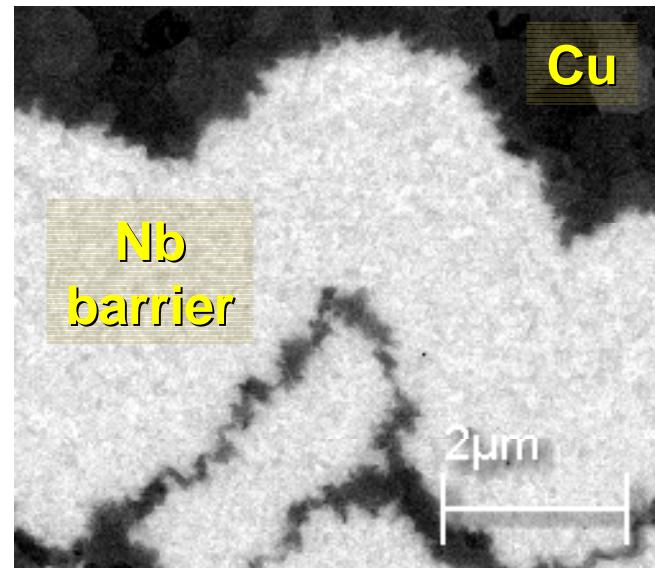
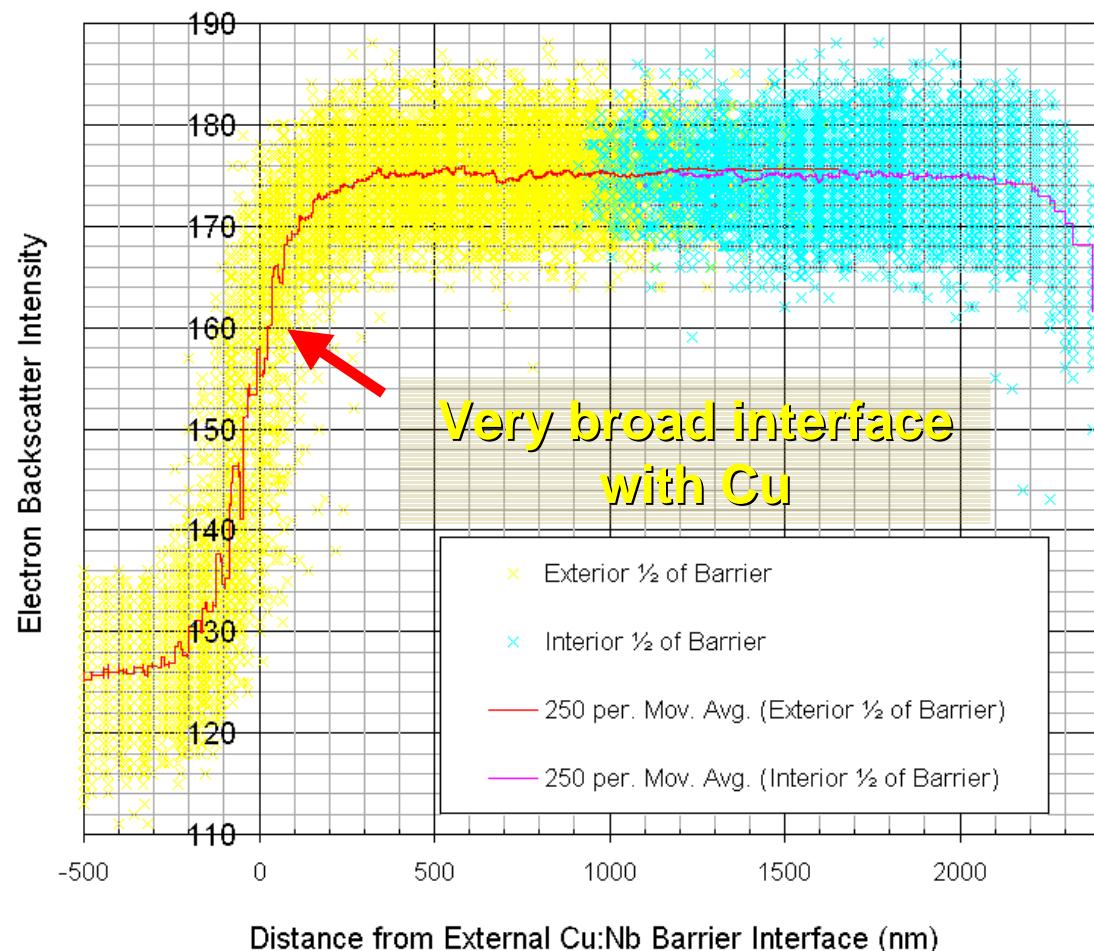


## Example III. IGC ITER Internal Sn Filament

**IGC**  
ADVANCED SUPERCONDUCTORS  
A DIVISION OF INTERMAGNETICS GENERAL CORPORATION

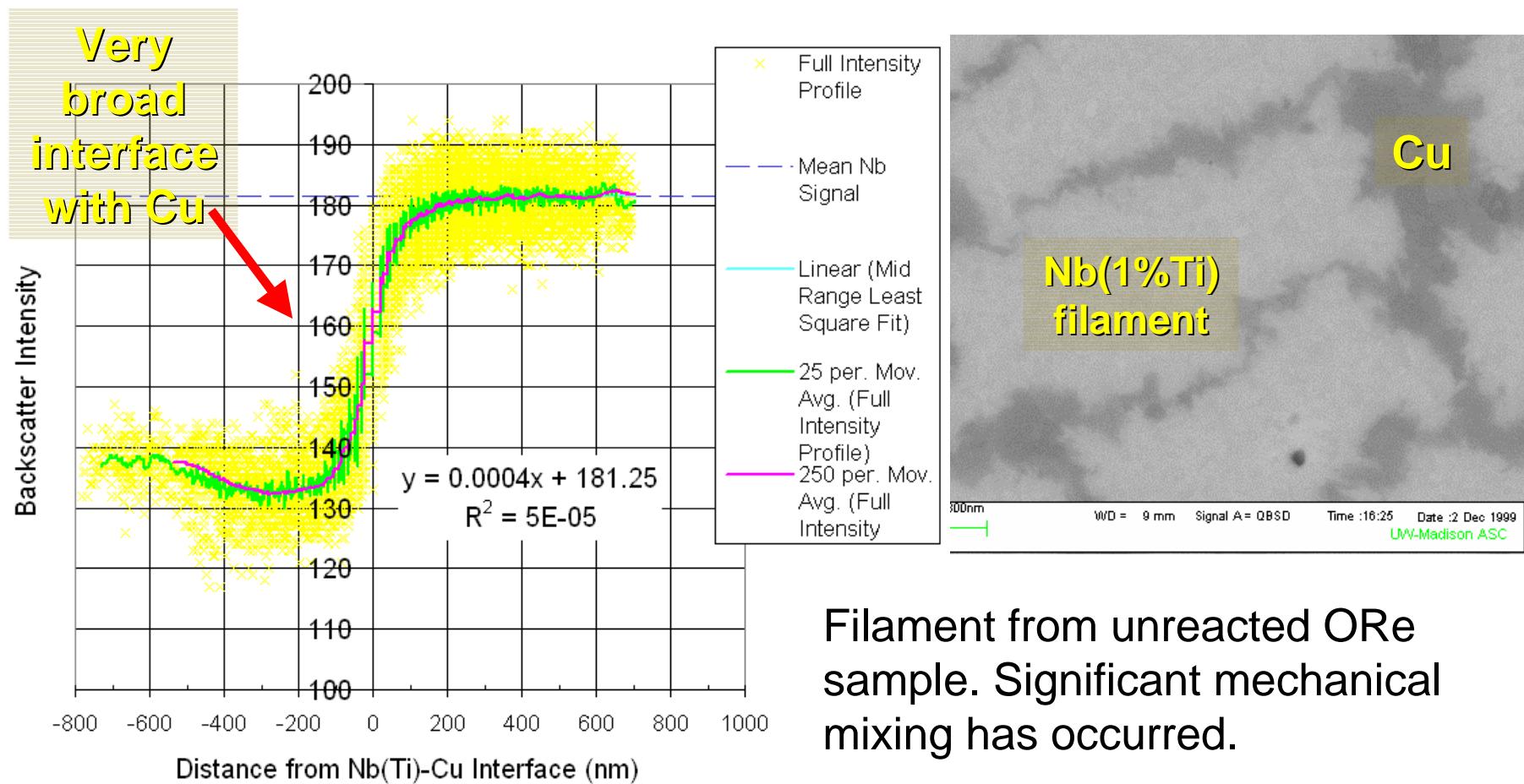


## *Example IVa. OI-ST high $J_c$ /Sn internal Sn Nb barrier before HT*



BES intensity across Nb barrier in ORE strand. For this image 1 pixel = 27.8 nm (compared to 10.3 nm for filament). Note broad transition due to mechanical mixing.

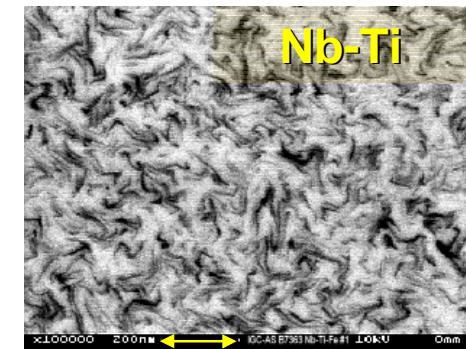
## *Example IVb. OI-ST high $J_c$ /Sn internal Sn filaments before HT*





## Summary

- Although 3000 A/mm<sup>2</sup> is a difficult target, Nb<sub>3</sub>Sn is still very inhomogeneous compared with optimized Nb-Ti which has:
  - chemical homogeneity and
    - microstructural homogeneity
- We are not close to the limit of what can be achieved in Nb<sub>3</sub>Sn.





# ***Is 3000 A/mm<sup>2</sup> Strand Possible?***

- We don't know!
  - Can we significantly change  $Q_{gb}$  in a composite?
    - ◆ We expect to be able to answer this soon.
- We do know it *is* possible in thin films (e.g. Dan Dietderich's work at LBNL)





# Additional Reading

- P. J. Lee, A. A. Squitieri and D. C. Larbalestier, "Nb<sub>3</sub>Sn: Macrostructure, Microstructure, and Property Comparisons for Bronze and Internal Sn Process Strands," *IEEE Transactions on Applied Superconductivity*, 10(1): 979-982, 2000. [Download PDF file \(1 MB, 4 pages\)](#)
  - <http://asc.wisc.edu/pubs/pub605.pdf>
- C. D. Hawes, P. J. Lee, and D. C. Larbalestier, "Measurement of the Critical Temperature Transition and Composition Gradient in Powder-In-Tube Nb<sub>3</sub>Sn Composite Wire," *IEEE Transactions on Applied Superconductivity*, 10(1): 988-991, 2000. [Download PDF file \(1.4 MB, 4 pages\)](#)
  - <http://asc.wisc.edu/pubs/pub625.pdf>
- M. T. Naus, P. J. Lee, and D. C. Larbalestier, "The Interdiffusion of Cu and Sn in Internal Sn Nb<sub>3</sub>Sn Superconductors," *IEEE Transactions on Applied Superconductivity*, 10(1): 983-987, 2000. [Download PDF file \(2.1 MB, 5 pages\)](#)
  - <http://asc.wisc.edu/pubs/pub624.pdf>
- P. J. Lee, J. R. Ruess and D. C. Larbalestier, "Quantitative Image Analysis of Filament Coupling and Grain Size in ITER Nb(Ti)<sub>3</sub>Sn Strand Manufactured by the Internal Sn Process," *IEEE Trans. Applied Superconductivity*, Vol. 7(2), pp. 1516-1519, 1997.
  - <http://asc.wisc.edu/pubs/pub407.pdf>
- P. J. Lee and D. C. Larbalestier, "Position Normalization as a Tool to Extract Compositional and Microstructural Profiles from Backscatter and Secondary Electron Images," to be published in the proceedings of *Microscopy & Microanalysis 2000*, Philadelphia, PA, 2000.
  - <http://asc.wisc.edu/pubs/pub643.pdf>

